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(54) **MULTI-ANALYTE OPTICAL COMPUTING SYSTEM**

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See application file for complete search history.

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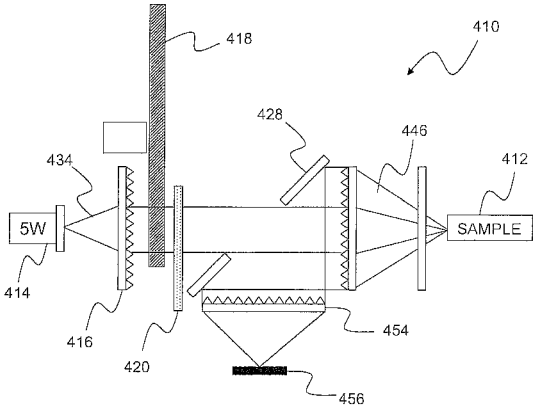
(57) **ABSTRACT**

The present subject matter relates to methods of high-speed analysis of product samples. Light is directed to a portion of a product under analysis and reflected from or transmitted through the product toward an optical detector. Signals for the detector are compared with reference signals based on a portion of the illuminating light passing through a reference element to determine characteristics of the product under analysis. The products under analysis may be stationary, moved by an inspection point by conveyor or other means, or may be contained within a container, the container including a window portion through which the product illuminating light may pass.

19 Claims, 4 Drawing Sheets



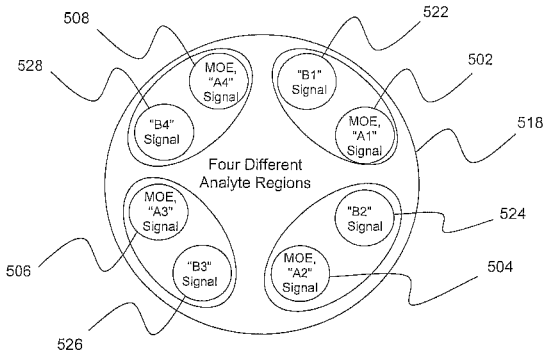


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FIG. 1

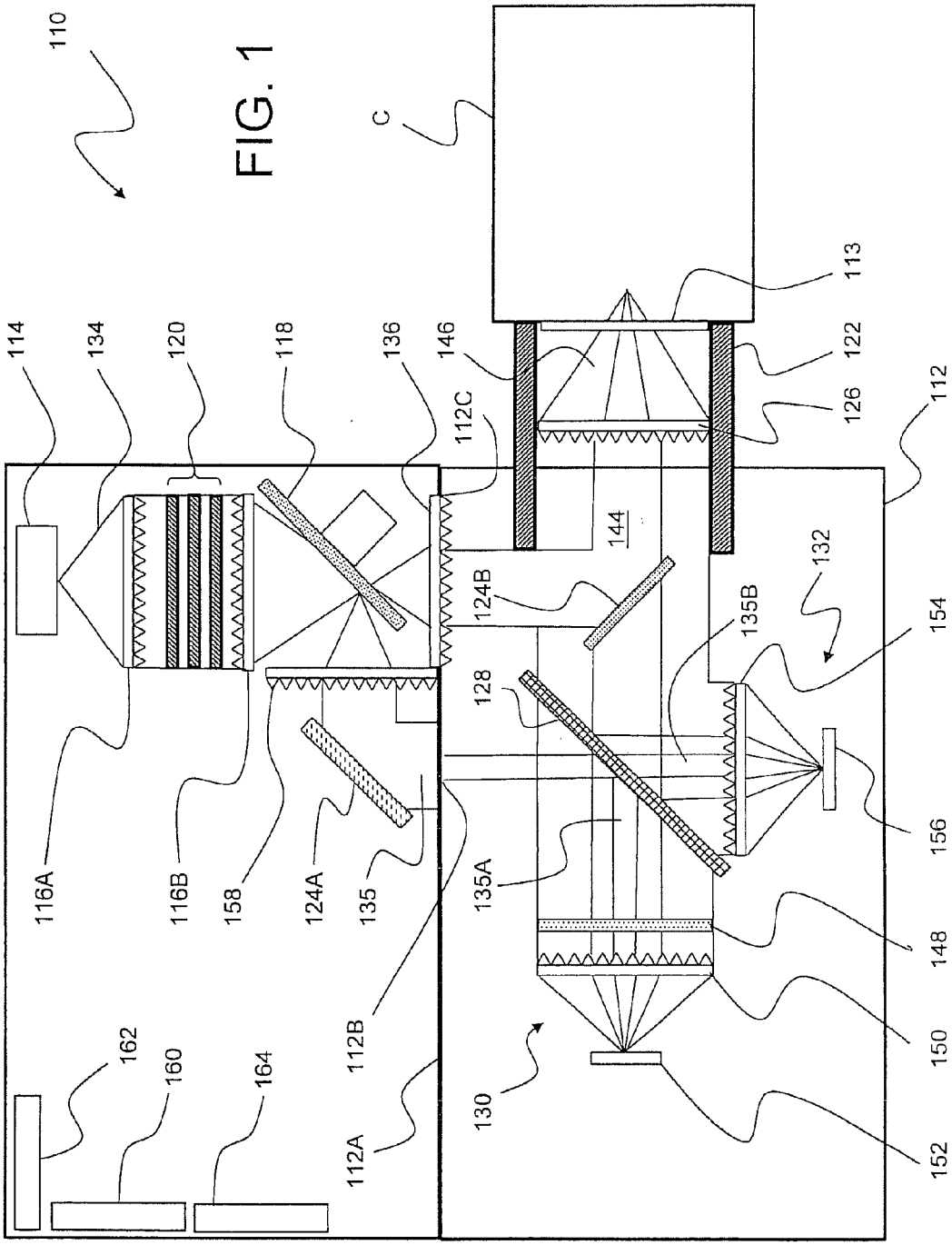
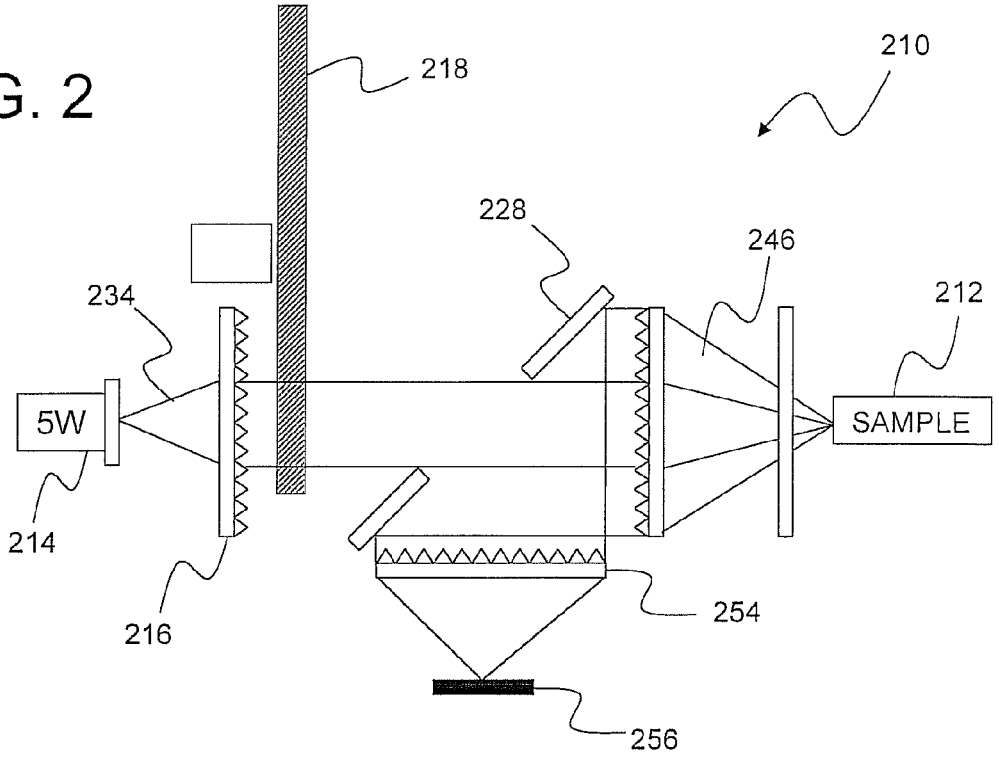

110
C
113
114
134
120
118
136
146
122
126
112C
112
144
132
124B
135B
154
128
116A
116B
158
124A
135
135A
156
148
150
162
160
164
112B
112A
130
152

FIG. 2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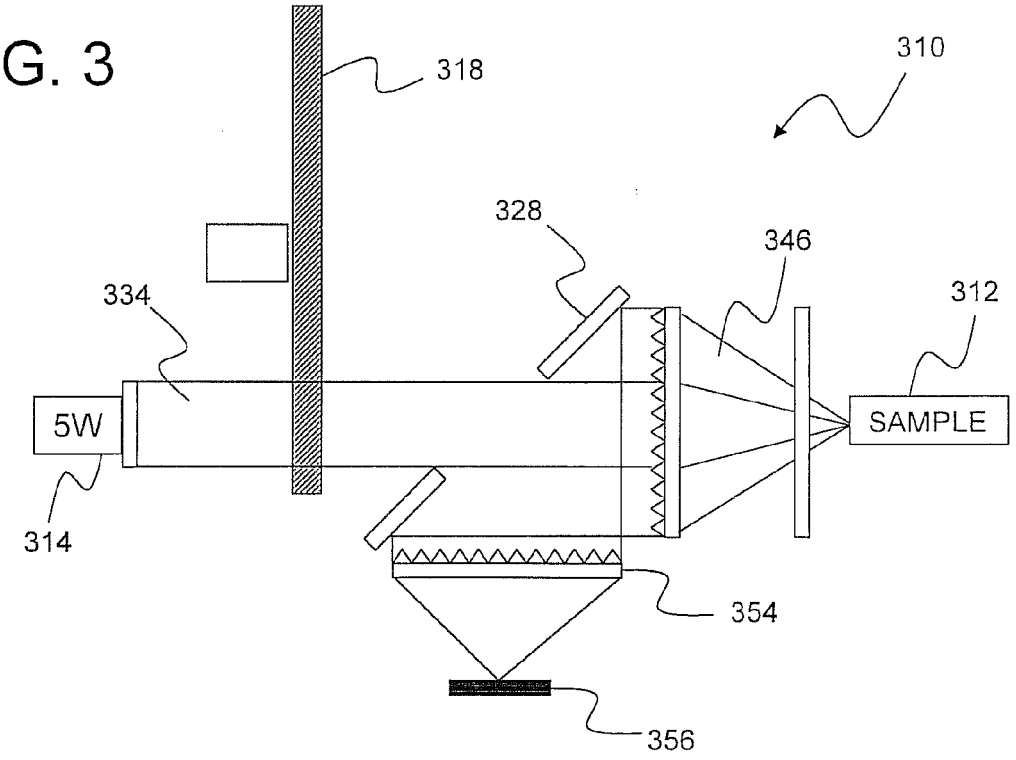
210
228
246
234
212
5W
SAMPLE
214
216
254
256

FIG. 3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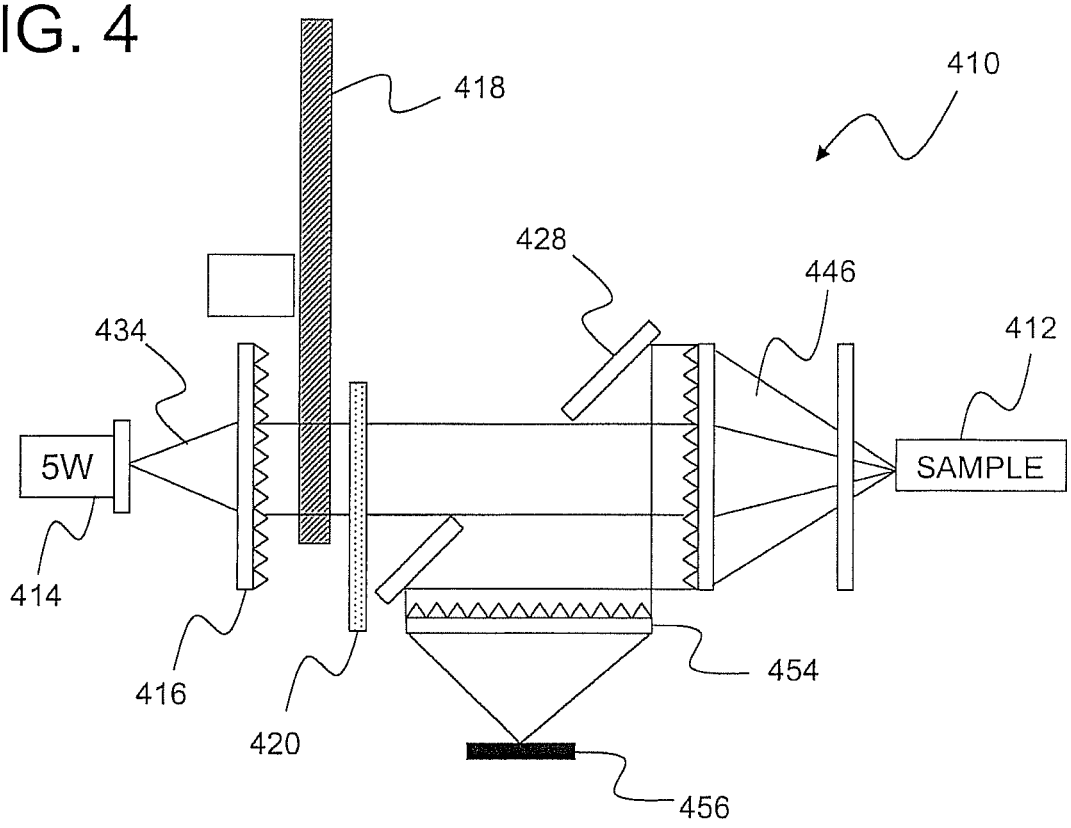
310
328
346
334
312
5W
SAMPLE
314
354
356

FIG. 4



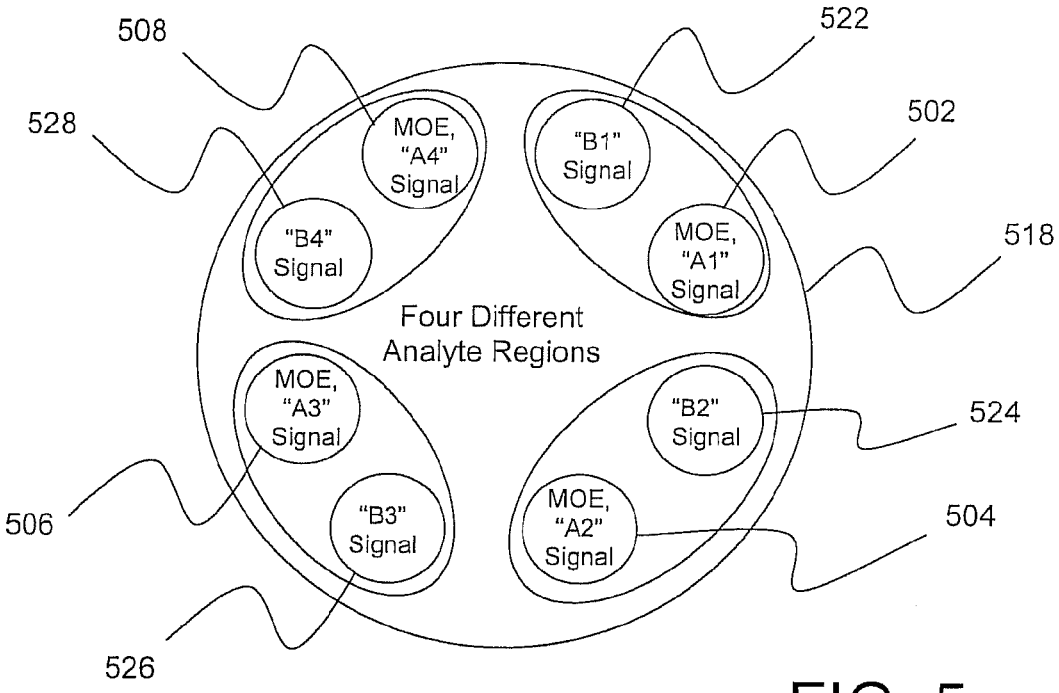

508
522
528
502
518
MOE, "A4" Signal
"B1" Signal
"B4" Signal
MOE, "A1" Signal
Four Different Analyte Regions
MOE, "A3" Signal
"B2" Signal
524
506
"B3" Signal
MOE, "A2" Signal
504
526

FIG. 5

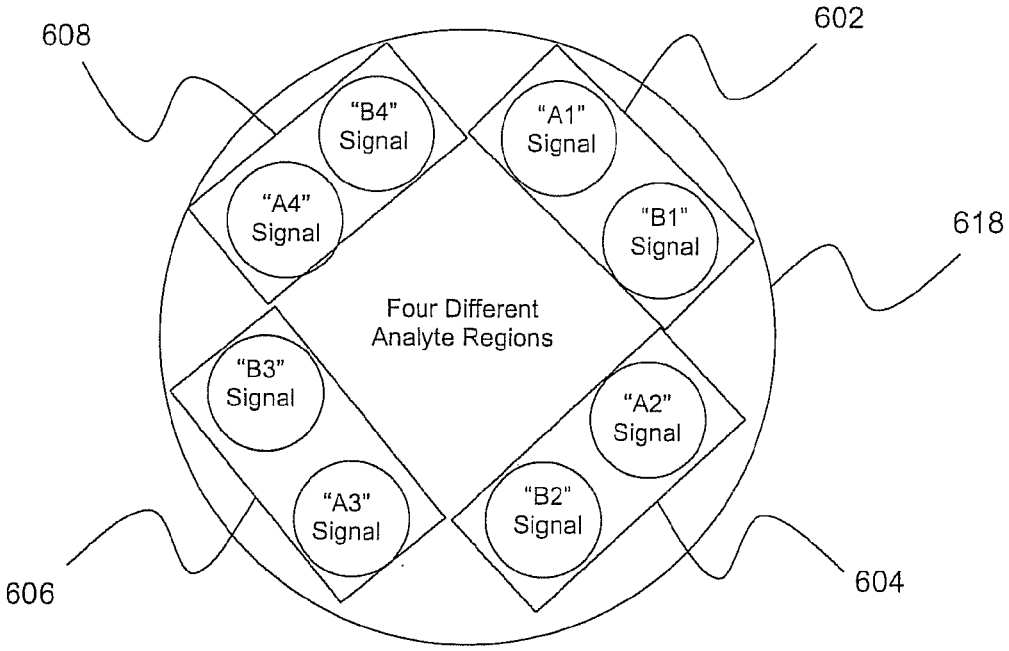

608
602
"B4" Signal
"A1" Signal
"A4" Signal
"B1" Signal
618
Four Different Analyte Regions
"B3" Signal
"A2" Signal
"A3" Signal
"B2" Signal
606
604

FIG. 6

MULTI-ANALYTE OPTICAL COMPUTING SYSTEM

This application claims priority under 35 USC 119(e) of Provisional Patent Application Ser. No. 60/856,191 filed Nov. 2, 2006, entitled "MULTI-ANALYTE OPTICAL COMPUTING SYSTEM," which is hereby incorporated by reference in its entirety.

FIELD OF THE INVENTION

The present subject matter relates to system design, fabrication and operation of multivariate optical elements. More particularly, the present subject matter relates to methodologies of using multivariate optical computing systems to illuminate a sample such that information about the sample can be analyzed from reflected or transmitted light in real time or near real time.

BACKGROUND OF THE INVENTION

Light conveys information through data. When light interacts with matter, for example, it carries away information about the physical and chemical properties of the matter. A property of the light, for example, its intensity, may be measured and interpreted to provide information about the matter with which it interacted. That is, the data carried by the light through its intensity may be measured to derive information about the matter. Similarly, in optical communications systems, light data is manipulated to convey information over an optical transmission medium, for example fiber optic cable. The data is measured when the light signal is received to derive information.

In general, measurement of light intensity may be difficult to convert to information due to contained interfering data. That is, several factors may contribute to the intensity of light, even in a relatively restricted wavelength range. It is often impossible to adequately measure the data relating to one of these factors since the contributions from other factors may be unknown.

It is possible, however, to derive information from light. An estimate may be obtained, for example, by separating light from several samples into wavelength bands and performing a multiple linear regression of the intensity of these bands against the results of conventional measurements of the desired information for each sample. For example, a polymer sample may be illuminated so that light from the polymer carries information regarding the sample's ethylene content. Light from each of several samples may be directed to a series of bandpass filters which separate predetermined wavelength bands from the light. Light detectors following the bandpass filters measure the intensity of each light band. If the ethylene content of each polymer sample is measured using conventional means, a multiple linear regression of ten measured bandpass intensities against the measured ethylene content for each sample may produce an equation such as:

$$y=a_0+a_1w_1+a_2w_2+\ldots+a_{10}w_{10} \quad \text{("Equation 1")}$$

where y is ethylene content, a_n are constants determined by the regression analysis, and w_n is light intensity for each wavelength band.

Equation 1 may be used to estimate ethylene content of subsequent samples of the same polymer type. Depending on the circumstances, however, the estimate may be unacceptably inaccurate since factors other than ethylene may affect the intensity of the wavelength bands. These other factors may not change from one sample to the next in a manner consistent with ethylene.

A more accurate estimate may be obtained by compressing the data carried by the light into principal components. To obtain the principal components, spectroscopic data is collected for a variety of samples of the same type of light, for example from illuminated samples of the same type of polymer. For example, the light samples may be spread into their wavelength spectra by a spectrograph so that the magnitude of each light sample at each wavelength may be measured. This data is then pooled and subjected to a linear-algebraic process known as singular value decomposition (SVD). SVD is at the heart of principal component analysis, which should be well understood by those of ordinary skill in this art. Briefly, however, principal component analysis is a dimension reduction technique, which takes in spectra with n independent variables and constructs a new set of eigenvectors that are linear combinations of the original variables. The eigenvectors may be considered a new set of plotting axes. The primary axis, termed the first principal component, is the vector, which describes most of the data variability. Subsequent principal components describe successively less sample variability, until only noise is described by the higher order principal components.

Typically, the principal components are determined as normalized vectors. Thus, each component of a light sample may be expressed as x_n, z_n, where x_n is a scalar multiplier and z_n is the normalized component vector for the n_{th} component. That is, z_n is a vector in a multi-dimensional space where each wavelength is a dimension. As should be well understood, normalization determines values for a component at each wavelength so that the component maintains it shape and so that the length of the principal component vector is equal to one. Thus, each normalized component vector has a shape and a magnitude so that the components may be used as the basic building blocks of all light samples having those principal components. Accordingly, each light sample may be described in the following format by the combination of the normalized principal components multiplied by the appropriate scalar multipliers:

$$x_1z_1+x_2z_2+\ldots+x_nz_n.$$

The scalar multipliers x_n may be considered the "magnitudes" of the principal components in a given light sample when the principal components are understood to have a standardized magnitude as provided by normalization.

Because the principal components are orthogonal, they may be used in a relatively straightforward mathematical procedure to decompose a light sample into the component magnitudes, which accurately describe the data in the original sample. Since the original light sample may also be considered a vector in the multi-dimensional wavelength space, the dot product of the original signal vector with a principal component vector is the magnitude of the original signal in the direction of the normalized component vector. That is, it is the magnitude of the normalized principal component present in the original signal. This is analogous to breaking a vector in a three dimensional Cartesian space into its X, Y and Z components. The dot product of the three-dimensional vector with each axis vector, assuming each axis vector has a magnitude of 1, gives the magnitude of the three dimensional vector in each of the three directions. The dot product of the original signal and some other vector that is not perpendicular to the other three dimensions provides redundant data, since this magnitude is already contributed by two or more of the orthogonal axes.

Because the principal components are orthogonal, or perpendicular, to each other, the dot, or direct, product of any principal component with any other principal component is zero. Physically, this means that the components do not interfere with each other. If data is altered to change the magnitude of one component in the original light signal, the other components remain unchanged. In the analogous Cartesian example, reduction of the X component of the three dimensional vector does not affect the magnitudes of the Y and Z components.

Principal component analysis provides the fewest orthogonal components that can accurately describe the data carried by the light samples. Thus, in a mathematical sense, the principal components are components of the original light that do not interfere with each other and that represent the most compact description of the entire data carried by the light. Physically, each principal component is a light signal that forms a part of the original light signal. Each has a shape over some wavelength range within the original wavelength range. Summing the principal components produces the original signal, provided each component has the proper magnitude.

The principal components comprise a compression of the data carried by the total light signal. In a physical sense, the shape and wavelength range of the principal components describe what data is in the total light signal while the magnitude of each component describes how much of that data is there. If several light samples contain the same types of data, but in differing amounts, then a single set of principal components may be used to exactly describe (except for noise) each light sample by applying appropriate magnitudes to the components.

The principal components may be used to accurately estimate information carried by the light. For example, suppose samples of a certain brand of gasoline, when illuminated, produce light having the same principal components. Spreading each light sample with a spectrograph may produce wavelength spectra having shapes that vary from one gasoline sample to another. The differences may be due to any of several factors, for example differences in octane rating or lead content.

The differences in the sample spectra may be described as differences in the magnitudes of the principal components. For example, the gasoline samples might have four principal components. The magnitudes x_n of these components in one sample might be J, K, L, and M, whereas in the next sample the magnitudes may be 0.94 J, 1.07K, 1.13 L and 0.86M. As noted above, once the principal components are determined, these magnitudes exactly describe their respective light samples.

Refineries desiring to periodically measure octane rating in their product may derive the octane information from the component magnitudes. Octane rating may be dependent upon data in more than one of the components. Octane rating may also be determined through conventional chemical analysis. Thus, if the component magnitudes and octane rating for each of several gasoline samples are measured, a multiple linear regression analysis may be performed for the component magnitudes against octane rating to provide an equation such as:

$$y=a_0+a_1x_1+a_2x_2+a_3x_3+a_4x_4 \quad \text{("Equation 2")}$$

where y is octane rating, a_n are constants determined by the regression analysis, and x_1, x_2, x_3 and x_4 are the first, second, third and fourth principal component magnitudes, respectively.

Using Equation 2, which may be referred to as a regression vector, refineries may accurately estimate octane rating of subsequent gasoline samples. Conventional systems perform regression vector calculations by computer, based on spectrograph measurements of the light sample by wavelength. The spectrograph system spreads the light sample into its spectrum and measures the intensity of the light at each wavelength over the spectrum wavelength range. If the regression vector in the Equation 2 form is used, the computer reads the intensity data and decomposes the light sample into the principal component magnitudes x_n by determining the dot product of the total signal with each component. The component magnitudes are then applied to the regression equation to determine octane rating.

To simplify the procedure, however, the regression vector is typically converted to a form that is a function of wavelength so that only one dot product is performed. Each normalized principal component vector z_n has a value over all or part of the total wavelength range. If each wavelength value of each component vector is multiplied by the regression constant a_n corresponding to the component vector, and if the resulting weighted principal components are summed by wavelength, the regression vector takes the following form:

$$y=a_0+b_1u_1+b_2u_2+\ldots+b_nu_n \quad \text{("Equation 3")}$$

where y is octane rating, a_0 is the first regression constant from Equation 2, b_n is the sum of the multiple of each regression constant a_n from Equation 2 and the value of its respective normalized regression vector at wavelength n, and u_n is the intensity of the light sample at wavelength n. Thus, the new constants define a vector in wavelength space that directly describes octane rating. The regression vector in a form as in Equation 3 represents the dot product of a light sample with this vector.

Normalization of the principal components provides the components with an arbitrary value for use during the regression analysis. Accordingly, it is very unlikely that the dot product result produced by the regression vector will be equal to the actual octane rating. The number will, however, be proportional to the octane rating. The proportionality factor may be determined by measuring octane rating of one or more samples by conventional means and comparing the result to the number produced by the regression vector. Thereafter, the computer can simply scale the dot product of the regression vector and spectrum to produce a number approximately equal to the octane rating.

In a conventional spectroscopy analysis system, a laser directs light to a sample by a bandpass filter, a beam splitter, a lens and a fiber optic cable. Light is reflected back through the cable and the beam splitter to another lens to a spectrograph. The spectrograph separates light from the illuminated sample by wavelength so that a detection device such as a charge couple detector can measure the intensity of the light at each wavelength. The charge couple detector is controlled by controller and cooled by a cooler. The detection device measures the light intensity of light from the spectrograph at each wavelength and outputs this data digitally to a computer, which stores the light intensity over the wavelength range. The computer also stores a previously derived regression vector for the desired sample property, for example octane, and sums the multiple of the light intensity and the regression vector intensity at each wavelength over the sampled wavelength range, thereby obtaining the dot product of the light from the substance and the regression vector. Since this number is proportional to octane rating, the octane rating of the sample is identified.

Since the spectrograph separates the sample light into its wavelengths, a detector is needed that can detect and distinguish the relatively small amounts of light at each wavelength. Charge couple devices provide high sensitivity throughout the visible spectral region and into the near infrared with extremely low noise. These devices also provide high quantum efficiency, long lifetime, imaging capability and solid-state characteristics. Unfortunately, however, charge couple devices and their required operational instrumentation are very expensive. Furthermore, the devices are sensitive to environmental conditions. In a refinery, for example, they must be protected from explosion, vibration and temperature fluctuations and are often placed in protective housings approximately the size of a refrigerator. The power requirements, cooling requirements, cost, complexity and maintenance requirements of these systems have made them impractical in many applications.

Multivariate optical computing (MOC) is a powerful predictive spectroscopic technique that incorporates a multi-wavelength spectral weighting directly into analytical instrumentation. This is in contrast to traditional data collection routines where digitized spectral data is post processed with a computer to correlate spectral signal with analyte concentration. Previous work has focused on performing such spectral weightings by employing interference filters called Multivariate Optical Elements (MOEs). Other researchers have realized comparable results by controlling the staring or integration time for each wavelength during the data collection process. All-optical computing methods have been shown to produce similar multivariate calibration models, but the measurement precision via an optical computation is superior to a traditional digital regression.

MOC has been demonstrated to simplify the instrumentation and data analysis requirements of a traditional multivariate calibration. Specifically, the MOE utilizes a thin film interference filter to sense the magnitude of a spectral pattern. A no-moving parts spectrometer highly selective to a particular analyte may be constructed by designing simple calculations based on the filter transmission and reflection spectra. Other research groups have also performed optical computations through the use of weighted integration intervals and acousto-optical tunable filters digital mirror arrays and holographic gratings.

The measurement precision of digital regression has been compared to various optical computing techniques including MOEs, positive/negative interference filters and weighted-integration scanning optical computing. In a high signal condition where the noise of the instrument is limited by photon counting, optical computing offers a higher measurement precision when compared to its digital regression counterpart. The enhancement in measurement precision for scanning instruments is related to the fraction of the total experiment time spent on the most important wavelengths. While the detector integrates or co-adds measurements at these important wavelengths, the signal increases linearly while the noise increases as a square root of the signal. Another contribution to this measurement precision enhancement is a combination of the Felgott's and Jacquinot's advantage, which is possessed by MOE optical computing.

While various implementations of Optical Analysis Systems have been developed to enhance measurement accuracy, no design has emerged that generally encompasses all of the desired characteristics as hereafter presented in accordance with the subject technology.

SUMMARY OF THE INVENTION

In view of the recognized features encountered in the prior art and addressed by the present subject matter, an improved methodology for high-speed processing and monitoring of a plurality of sample portions of products has been developed. In accordance with an exemplary configuration of the present subject matter, products may be moved past an inspection point while illuminating at least one portion of the sample product with a spectral-specific light through an optic window. The optical window may be configured to focus the spectral-specific light onto a sample portion at the inspection point. In an exemplary embodiment, the sample products may include pharmaceutical products.

In one representative configuration, light reflecting from the sample, which contains information about the sample, passes through a chopper wheel and onto a detector. As the chopper wheel rotates, the light passes through different elements depending on the orientation of the wheel at a given time. Hence the signal at the detector changes sequentially as light passes through the different multivariate optical elements or reference elements or the light is blocked (where the spokes on the chopper wheel block the light). A different signal is produced for each element. These signals are used to determine at high speed one or more selected properties of the portion as it moves past the inspection point based upon the detector output.

In an exemplary arrangement, the sample portions may comprise pharmaceutical tablets, trays or other containers of powders, or partially- or fully-enclosed sample containers at least partially transparent to light focused onto the sample portion. In an exemplary configuration, sample portions may be moved past the inspection point at a rate between about one portion/second and about five portions/second, with monitoring occurring in real-time at high speed.

In accordance with aspects of certain other embodiments of the present subject matter, a method of high-speed processing and monitoring includes moving a product past an inspection point and illuminating at least a portion of the product with a light. Light reflecting from the sample, which contains information about the sample, passes through a chopper wheel and onto a detector. As the chopper wheel rotates, the light passes through different elements depending on the orientation of the wheel at a given time. Hence the signal at the detector changes sequentially as light passes through the different multivariate optical elements or reference elements or the light is blocked where the spokes on the chopper wheel block the light. A different signal is produced for each element. These signals are used to determine, at high speed, one or more selected properties of the sample portion as it moves past the inspection point based upon the detector output. The product in this aspect may be, for example, a pharmaceutical tablet or powder, a food material, a chemical, a liquid, a gas, an emulsion, a solution, and/or a mixture.

Additional objects and advantages of the present subject matter are set forth in, or will be apparent to, those of ordinary skill in the art from the detailed description herein. Also, it should be further appreciated that modifications and variations to the specifically illustrated, referred and discussed features and elements hereof may be practiced in various embodiments and uses of the invention without departing from the spirit and scope of the subject matter. Variations may include, but are not limited to, substitution of equivalent means, features, or steps for those illustrated, referenced, or discussed, and the functional, operational, or positional reversal of various parts, features, steps, or the like.

Still further, it is to be understood that different embodiments, as well as different presently preferred embodiments, of the present subject matter may include various combinations or configurations of presently disclosed features, steps, or elements, or their equivalents (including combinations of

features, parts, or steps or configurations thereof not expressly shown in the figures or stated in the detailed description of such figures). Additional embodiments of the present subject matter, not necessarily expressed in the summarized section, may include and incorporate various combinations of aspects of features, components, or steps referenced in the summarized objects above, and/or other features, components, or steps as otherwise discussed in this application. Those of ordinary skill in the art will better appreciate the features and aspects of such embodiments, and others, upon review of the remainder of the specification.

BRIEF DESCRIPTION OF THE DRAWINGS

A full and enabling disclosure of the present invention, including the best mode thereof to one skilled in the art, is set forth more particularly in the remainder of the specification, including reference to the accompanying figures, in which:

FIG. **1** is schematic plan view of a real time measurement system according to the general concept of the present technology;

FIG. **2** is a schematic view of a real time measurement system according to the present subject matter illustrating a focusing lens at the illumination source;

FIG. **3** is a schematic view of a real time measurement system according to the present subject matter illustrating collimated light at the illumination source;

FIG. **4** is a schematic view of a real time measurement system according to the present subject matter illustrating a set of common spectral elements for all the measurements;

FIG. **5** is a schematic view of the chopper wheel containing several multivariate optical elements (A) and reference elements (B); and

FIG. **6** is a schematic view of a chopper wheel illustrating the inclusion of separate spectral elements for each of four analytes being measured.

Repeat use of reference characters throughout the present specification and appended drawings is intended to represent same or analogous features or elements of the invention.

DETAILED DESCRIPTION OF THE INVENTION

As discussed in the Summary of the Invention section, the present subject matter is particularly concerned with an improved methodology for high-speed processing and monitoring of a plurality of sample product portions.

Selected combinations of aspects of the disclosed technology correspond to a plurality of different embodiments of the present invention. It should be noted that each of the exemplary embodiments presented and discussed herein should not insinuate limitations of the present subject matter. Features or steps illustrated or described as part of one embodiment may be used in combination with aspects of another embodiment to yield yet further embodiments. Additionally, certain features may be interchanged with similar devices or features not expressly mentioned which perform the same or similar function.

As used herein, the term "light" is broadly used to mean any form of radiation or radiative energy including, but not limited to, visible light or light in the infrared region. "Light" is also referred to herein as a light signal, a light beam, a light ray and the like to mean any form of radiative energy in the electromagnetic spectrum. Similarly, the term "transmission" can mean transmission of radiative energy onto a surface of a sample; penetration, however slight, into a sample such as a particulate sample or opaque fluid sample; or passage through a sample.

As used herein, the sample being evaluated can be a solid or a fluid including, but not limited to, a powder, a pharmaceutical powder mixed with lactose and other excipient materials, a chemical, a polymer, a petroleum product, a solution, a dispersion, an emulsion and combinations of these solids and fluids.

Reference will now be made in detail to the presently preferred embodiments of the subject optical computing system. Referring now to the drawings, FIG. **1** illustrates an optical analysis system **110** generally depicting the concept of the present subject matter. As shown in FIG. **1**, optical analysis system **110** broadly includes a housing **112**, an illumination or light source **114**, a chopper wheel **118**, one or more spectral elements **120**, a focusing lens **126**, a beam splitter **128**, a first detector **130** including a multivariate optical element **148**, and a second detector **132**. Optical analysis system **110** further includes representatively illustrated electrical connection **160**, pressurization sensor **162** and purge gas assembly **164**. These representatively illustrated components are well understood by those of ordinary skill in the present art and, therefore, further description is not deemed necessary to understand and practice these aspects of the present subject matter.

With more particular reference to FIG. **1**, illumination source **114** provides a light **134**, which passes through a collecting Fresnel lens **116**A and into and through spectral element(s) **120**. In an exemplary configuration, illumination source **114** may be rated for at least about 10,000 hours of operation, which alleviates a need for redundant illumination sources though such redundant sources may be provided if desired. Further, in the illustrated exemplary configuration, the collecting Fresnel lens **116**A is sized to be about 1.5 square inches and is spaced about 0.6 inches from the illumination source **114**. Those of ordinary skill in the art will appreciate that these dimensions can be adjusted according to particular system requirements and are thus not meant as limitations of the present subject matter.

As further shown in FIG. **1**, light **134** passes through spectral elements **120**, which filter out undesired wavelengths to define a desired spectral region in order to target a particular chemical material of interest. In an exemplary configuration the spectral region may correspond to, 1500-2000 nm. Light **134** is focused by focusing Fresnel lens **116**B, which, in an exemplary configuration may also be sized to be about 1.5 square inches and spaced about 1 inch from the chopper wheel **118**. As shown, the chopper wheel **118** reflects a portion of light **134** as a calibration or reference light **135** and passes another portion as transmitted light **144**.

Calibration light **135** is collimated by lens **158** before reflecting from a first mirror **124**A through an adjustable aperture **112**B in a bulkhead **112**A of the housing **112**. Aperture **112**B is adjustable to control the amount of the calibration light **135** passing through the aperture. Finally, calibration light **135** impinges on beam splitter **128** thereby sending a portion **135**A of calibration light **135** to the first MOE detector **130** and a portion **135**B of calibration light **135** to the second or baseline detector **132**.

With further reference to FIG. **1**, it may be seen that transmitted light **144** passes from the chopper wheel **118** into a collimating Fresnel lens **136**, which in this exemplary configuration is sized to be about 1.5 square inches and is spaced about 0.6 inches from the chopper wheel **118**. Transmitted light **144** passes through another adjustable aperture **112**C in the bulkhead **112**A and impinges upon a second mirror **124**B, which directs transmitted light **144** toward a sample in a container C. In an exemplary configuration, container C may correspond to a mixing vat or blender. Those of ordinary skill

in the art will appreciate that "container" C could correspond to a conveyor belt or other device for holding or transporting the sample and is thus not limited to an enclosed container.

As further illustrated in FIG. **1**, transmitted light **144** is focused by focusing Fresnel lens **126**, which in this exemplary configuration may be round and about 15/16 inches in diameter and is adjustable with an inner tube **122**. Also in this exemplary configuration, lens **126** may be positioned about 0.6 inches from an outer surface of the container C. As shown, transmitted light **144**, now focused by Fresnel lens **126**, passes through a transmissive window **113**. In an exemplary configuration transmissive window **113** may be approximately 1 inch in diameter and include an anti-reflective (AR) coating disposed on one or both sides of the window **113**. The AR coating ensures that the chemical process in the container C does not interfere with the measuring process of optical analysis system **110**. Thus, transmitted light **144** enters the container C and reflects from the sample as carrier light **146**. The sample can be a moving mixture such as aspirin and an excipient being blended in real time, or a plurality of tablets passing by on a conveyor belt at high speed.

Referring further to FIG. **1**, it will be seen that carrier light **146** is directed by the tube **122** in a direction of the first detector **130**. Eventually, carrier light **146** impinges on beam splitter **128** so that a portion of carrier light **146** passes in a direction of detector **132** for baselining with portion **135**B of calibration light **135**. Another portion of carrier light **146** passes through MOE **148**, which as noted above, has been selected for the chemical of interest based on the various components of the system **110**. Finally, that portion of carrier light **146**, having passed through the MOE **148**, is focused by lens **150** and received by the detector **152**. As described above, the two signals collected by the detectors **152** and **156** can be manipulated, e.g., mathematically, to extract and ascertain information about the sample carried by the carrier light **146**.

In accordance with the present subject matter, further exemplary embodiments of optical computing systems based on the optical computational method previously described with reference to FIG. **1** are described herein after with reference to FIGS. **2-6**. In general, these further embodiments differ from that described in FIG. **1** in that they employ a single detector with a moving holder, such as a chopper wheel, containing several multivariate optical elements and reference materials. In accordance with these embodiments of the present subject matter, the chopper wheel rotates so that the various elements pass in front of the detector in sequence enabling the use of several optical elements in the same unit. The chopper wheel containing the multivariate optical elements (MOEs) and spectral elements must rotate fast enough to compensate for sample movement and various light fluctuations. Each multivariate optical element and associated reference element is designed for measurement of a specific property. Because the spectral elements and multivariate optical elements are used as a combination for a given analyte, several configurations are presented each illustrating variations in spectral element installation.

Each of FIGS. **2-6** illustrate further embodiments of optical computing systems operating in accordance with the general concept of the optical computing system illustrated in FIG. **1**. With reference to FIG. **2** a first exemplary embodiment of an optical computing system **210** constructed in accordance with the present subject matter and employing a single detector **256** will be described. As illustrated in FIG. **2**, lens **216** is configured to collect light **234** emitted by illumination source **214** and to direct it towards sample **212** by way of chopper wheel **218** and mirror **228**. Reflected light **246** from sample **212** reflects from mirror **228** toward detector **256** by way of focusing lens **254**. In an exemplary configuration, mirror **228** may be oval, however, other shapes, e.g., round, may also be employed. Further different mirror configuration may include one where illuminating light passes through an outer annual region to illuminate the sample as opposed to the central portion illustrated, while the turning mirror returns the inner portion of the light toward the detector.

FIG. **3** illustrates a configuration identical to that of FIG. **2** except that illumination source **314** produces collimated light. Remaining reference numerals in FIG. **3** correspond directly to those of FIG. **2** except for being designated in the **300**'s series as opposed to the **200**'s series of FIG. **2** and will thus not be further described. It should be appreciated that as illumination source **314** produces collimated light, and equivalent of FIG. **2**,'s lens **216** is not required.

The configurations illustrated in FIGS. **2** and **3** employ spectral elements located on their respective chopper wheels **218**, **318** as illustrated by rectangles **602**, **604**, **606**, **608** on chopper wheel **618** of FIG. **6**. As illustrated in FIG. **6**, spectral elements within rectangle **602** are employed for a first analyte, while spectral elements within rectangles **604**, **606**, and **608** are employed for second, third, and forth analytes, respectively. If the same spectral elements are used for all analytes, then a common spectral element **420** can be employed as shown in FIG. **4**. It should be appreciated by those of ordinary skill in the art that the components illustrated in FIG. **4** are generally identical to those of FIGS. **2** and **3** and are thus identified with equivalent but **400**'s series numbers. Those of ordinary skill in the art will also appreciate that the embodiment illustrated in FIG. **4** may also be modified in a manner like that of the embodiment of FIG. **3** to provide collimated light in place of the illustrated light source **414** and collimating lens **416**.

FIG. **5** illustrates an exemplary configuration of a chopper wheel **518** containing multivariate elements **502**, **504**, **506**, **508** and reference elements **522**, **524**, **526**, **528** for each of four independent analytes. FIG. **6** illustrates an opposite side view of the same chopper wheel shown in FIG. **5** with the spectral elements indicated as rectangles. In this example, there are separate spectral element units for each of four analytes.

In accordance with the single detector system of the present subject matter, "A" and "B" signals for each multivariate optical element are collected in sequence. As illustrated in FIG. **5**, the signal sequence produced by chopper wheel **518** (FIG. **5**) would correspond to Analyte **1**-B signal, Analyte **1**-A signal, Analyte **2**-B signal, Analyte **2**-A signal, Analyte **3**-B signal, Analyte **3**-A signal, Analyte **4**-B signal, Analyte **4**-A signal, Analyte **1**-B signal, and so forth. Multivariate optical element signal and reference signal calculations conducted on these signals are performed in the time domain. This is important to make sure that the Analyte **1** multivariate optical element (A) signal is compared to the Analyte **1** reference (B) signal.

The functionality of MOC system **110** (FIG. **1**) and the single detector embodiments as described above allow for the collection of the entire spectral range of testing simultaneously. This fact is notably different than either a system based on either a scanning lamp or detector system or a discrete diode array detection system. The ability to monitor over the complete spectral range of interest opens up a redefinition of the term "real-time" measurement and analysis. For instance, true real-time process measurements are possible.

In accordance with present disclosure, "real time" refers to obtaining data without delays attendant to collecting samples

or delays due to lengthy computer processing of measurement signals. In accordance with the subject matter described herein, process data can be obtained in an instantaneous or near-instantaneous manner through using the disclosed measurement techniques to directly monitor materials of interest while such materials are undergoing process steps. Long delays due to processing of measurement signals are avoided by optically processing the light as it is reflected from the material(s) of interest.

Although specific examples disclosed herein describe monitoring blending of a powdered material and examining solid tablets, the general concept can be extended to other phases. The present system can be utilized in analyzing solids, solutions, emulsions, gases, and dispersions, for example. In addition, while exemplary embodiments discussed herein use reflectance measurements, measurements in a transmission or transflectance mode would also be appropriate and is fully contemplated by the present disclosure.

One of ordinary skill in the art will recognize that differing applications may require modifications and alterations to certain components in order to take full advantage of the presently-disclosed systems, all of which adjustments would be well within the capability of those of ordinary skill in the art given an understanding of the present disclosure. For instance, more diffusion of light has been observed in solid powders relative to liquids; accordingly, different lenses may be needed when a liquid is monitored in order to account for such variations and achieve more accurate measurements.

The presently-disclosed technology can be applied to real-time measurements for a range of industrial applications. These include, but are not limited to, monitoring of the blending of pharmaceutical powders, including excipients, additives, and active pharmaceutical materials; blending of other powders, including food and chemicals; monitoring dispersions and bi-phasic mixtures (such as insulin, emulsions); and oil and gas applications, including analyzing water content in oil, or oil content in water.

Inclusion of a transmissive window provides physical separation between the measuring device and the process or material being tested. Therefore, this window allows for in-line measurement and/or non-invasive measurement of parameters such as chemical functionality, including alcohol content of petroleum fractions or tackifier resins. Environmental applications are also conceivable, such as stack gas analysis, including measurement of NOx, SOx, CO, CO2, or other gases in a gas stream; wastewater analysis and treatment monitoring; and hazardous substance monitoring applications such as mercury vapor detection.

As noted above, MOC technology in accordance with present disclosure can be used to monitor a wide variety of materials as the materials are subjected to different processes. For instance, the mixing of powders can be monitored. As materials are blended, the existing art does not allow for continuous, real-time, in-line measurement. Current limitations are the result of several factors including: moving of the powders being measured during the course of data acquisition and the need to connect analytical equipment to the measurement point using fiber optic cables. This optical analysis system is designed to allow for instantaneous measurement using a measurement point located on the vessel.

Other embodiments of the present subject matter provide for real time measurement of flowing materials. In such embodiments, sampling window(s) may be located on a pipe or vessel such that interrogating illumination can be applied to the material. For instance, a port could be included on a pipe to allow for sampling of the material inside the pipe. The window may be positioned directly on the pipe, or on a small diversion away from the main flow path, as appropriate under the circumstances. Such embodiments could also include sampling of vapor systems within a stack to monitor combustion gases or flowing process stream such as water containing other materials.

Still further embodiments of the present subject matter provide real time measurement of materials in containers, such as vials or bins where the container is either at least partially open to the outside environment or transmissive to the sampling illumination. Such containers could be stationary or in motion. A container could also include a conveyor or trough carrying material. Typical applications could include the monitoring the progress of a chemical reaction or the content of samples moving past a measurement location.

The present subject matter may be better understood from the following tests and examples. In a first example, a breadboard system was constructed and used to test a mixture of powders. The first system components included a 20W Gilway lamp and employed a 5 mm deuterium oxide (D_2O) and 5 mm Germanium spectral elements. A fiber optic probe was employed as an optical window while an InAr detector was used for measurements. For this example, a powdered sample with a known composition was placed in a dish and the fiber optic probe was placed in contact with the powder. The output of the detectors was monitored and recorded.

In a second example, a system was constructed and used to make static measurements on aspirin/lactose. The second example system employed components identical to those of the first sample except for the detector where a PbS detector from New England Photoconductor was employed instead of the InAr detector. For this example, a powdered sample with a known composition was placed in a dish and the system light beam was focused on the powder. The output of the detectors was monitored and recorded. Aspirin/lactose samples covering the range of 100% aspirin to 100% lactose were tested.

In a third example, a system similar to that employed in the second example was employed except that a sapphire window was employed to gain access to the test material light borne data. In this example, Aspirin/Lactose testing was performed using a mixer bowl containing lactose and the system measured as aspirin was added to the system and mixed. Specifically, lactose powder was placed in the bowl of a mixer and the measurement system was attached the bowl using a Swagelok® brand fitting. A sapphire window was used to contain the powder in the bowl and allow the system to interrogate the powder. With the mixer turning, known amounts of aspirin were added and the system output signal was monitored and recorded. Aspirin was added in several allotments to about 37% final aspirin concentration.

Although the present subject matter has been described in such a way as to provide an enabling disclosure for one skilled in the art to make and use the invention, it should be understood that the descriptive examples are not intended as limitations of the subject matter as described herein and shown in the Figures. For instance, the housing can be shaped as a square, an oval, or in a variety of other shapes. Further, a variety of light sources can be substituted for those described above. It is intended to claim all such changes and modifications as fall within the scope of the appended claims and their equivalents. Thus, while exemplary embodiments of the invention have been shown and described, those skilled in the art will recognize that changes and modifications may be made to the foregoing examples without departing from the scope and spirit of the present subject matter.

What is claimed is:

1. A method for high-speed analysis of product samples comprising:

transmitting an illumination light through a analyte multivariate optical element to form an analyte light, the multivariate optical element being positioned on a chopper wheel;

illuminating a sample at an inspection point with the analyte light;

transmitting the illumination light through a reference optical element to form a reference light different from the analyte light, the reference optical element being positioned on the chopper wheel and configured to correspond to the multivariate optical element;

illuminating the sample with the reference light;

providing a light sensitive detector, the detector producing an output signal based on received light;

directing light from the sample toward the light sensitive detector, the light from the sample carrying information about the sample; and

analyzing output signals produced by the detector, the analysis being based on a comparison of an output signal produced by the analyte light and an output signal produced by the reference light.

2. The method of claim 1, wherein the sample is at least one of a pharmaceutical tablet, a pharmaceutical powder, a food material, a chemical, a liquid, a gas, an emulsion, a solution, or a mixture thereof.

3. The method of claim 1, wherein the sample is a powder mixture in a closed container, the container being at least partially transparent to the illumination light.

4. The method of claim 1, further comprising:

moving the sample past the inspection point.

5. The method of claim 1, wherein directing light from the sample comprises directing light reflected from the sample.

6. The method of claim 1, wherein directing light from the sample comprises directing light transmitted through the sample.

7. The method of claim 1, wherein illuminating the sample with the analyte light comprises illuminating the sample with a spectral-specific light.

8. The method of claim 7, further comprising:

illuminating the sample through an optic window, the optic window being configured to focus the spectral-specific light onto a sample portion at the inspection point.

9. The method of claim 1, wherein the sample comprises a plurality of discrete portions.

10. The method of claim 9, wherein the plurality of discrete portions are disposed in closed containers, the containers at least partially transparent to the spectral-specific light.

11. The method of claim 1, wherein:

transmitting the illumination light through the analyte multivariate optical element comprises directing the illumination light through a plurality of multivariate optical elements; and

transmitting the illumination light through the reference optical element comprises directing the illumination light through a plurality of reference optical elements.

12. The method of claim 11, wherein the illumination light is transmitted alternately through differing multivariate optical elements and corresponding reference optical elements in sequence.

13. The method of claim 1 further comprising determining a specific property of an analyte in the sample associated with the multivariate optical element and the reference optical element.

14. The method of claim 13 wherein the analyte includes a gasoline.

15. The method of claim 14 wherein the specific property of the analyte is an octane rating of the gasoline.

16. A system for high-speed analysis of samples comprising:

an illumination source to provide an illumination light to at least a sample at an inspection point, the sample comprising at least one analyte;

a light sensitive detector to produce an output based on a received light;

a chopper wheel comprising:

a multivariate optical element placed between the illumination source and the sample; and

a reference optical element placed between the illumination source and the sample, wherein the reference optical element corresponds to the multivariate optical element; and

an optical element to direct light from the sample to the light sensitive detector;

wherein the multivariate optical element and the reference optical element are selected for measurement of a specific property of the analyte, and for illuminating the sample with an analyte light different from a reference light.

17. The system of claim 16 further comprising a plurality of multivariate optical elements, each of the plurality of optical elements associated with a reference optical element, the multivariate optical element and the reference optical element selected to measure a specific property of the analyte.

18. The system of claim 16 wherein the analyte comprises at least a gasoline sample.

19. The system of claim 18 wherein the specific property is an octane rating of the gasoline sample.

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